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PART XIII DETERMINATION OF ARSENIC

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METHODS FOR CHEMICAL ANALYSIS OF STEELS

PART XIII DETERMINATION OF ARSENIC

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METHODS FOR CHEMICAL ANALYSIS OF STEELS

PART XIII DETERMINATION OF ARSENIC

0. FOREWORD

0.1 This Indian Standard (Part XIII) was adopted by the Indian Standards Institution on 22 February 1982, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 Earlier, determination of arsenic in iron and steel was covered in IS : 1546-1960*. The Sectional Committee responsible for the revision of this standard decided to publish this as a part of IS : 228† for comprehensive analysis of steel. However this method could also be applied for the determination of arsenic in iron. With the publication of this part, IS : 1546-1960* will be withdrawn.

0.3 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960‡.

1. SCOPE

1.1 This standard (Part XIII) prescribes the method for determination of arsenic in steel.

2. QUALITY OF REAGENTS

2.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977§) shall be used.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

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*Method for determination of arsenic in iron and steel.

†Methods for chemical analysis of steels.

‡Rules for rounding off numerical values (*revised*).

§Specification for water for general laboratory use (*second revision*).

3. GENERAL

3.1 Preparation of Filter Paper Pulp Pad — For the filtration of arsenic precipitate, filter paper pulp pad shall be used. It shall be prepared by shaking small pieces of filter paper with a mixture of one part of saturated solution of bromine in concentrated hydrochloric acid (rd = 1·16) and seven parts of water. When sufficiently disintegrated, the mixture shall be heated on the steam bath for 30 minutes and diluted with an equal volume of water, shaken and bottled. A filter pad shall be made of this pulp and washed well with water.

4. DETERMINATION OF ARSENIC IN STEEL

4.1 Outline of the Method — Arsenic which in steel exists mainly as iron arsenide ($FeAs_2$) is converted into arsenic acid (H_3AsO_4) when the alloy is dissolved in a mixture of sulphuric acid and nitric acid. Arsenic acid is reduced quantitatively in acid medium by sodium hypophosphite to metallic arsenic and the metal is then oxidized by a measured excess of standard iodine solution to pentavalent arsenic. The excess iodine is back titrated with standard arsenious oxide solution.

4.2 Reagents

4.2.1 *Electrolytic Copper*

4.2.2 *Dilute Sulphuric Acid* — 1 : 3 (*v/v*).

4.2.3 *Concentrated Nitric Acid* — rd = 1·42 (conforming to IS : 264-1976*).

4.2.4 *Concentrated Hydrochloric Acid* — rd = 1·16 (conforming to IS : 265-1976†).

4.2.5 *Syrupy Phosphoric Acid* — 85 percent.

4.2.6 *Mixed Acids* — Add 200 ml of syrupy phosphoric acid, 100 ml of concentrated hydrochloric acid, and 75 ml of concentrated nitric acid to 400 ml of water. Mix well and cool. Add carefully 150 ml of dilute sulphuric acid (1 : 3) and make up to one litre.

4.2.7 *Dilute Nitric Acid* — 1 : 50 (*v/v*).

4.2.8 *Potassium Permanganate* — solid.

4.2.9 *Sulphurous Acid Solution* — Prepare a saturated solution of sulphur dioxide in water.

4.2.10 *Sodium Hypophosphite* — solid.

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*Specification for nitric acid (*second revision*).

†Specification for hydrochloric acid (*second revision*).

4.2.11 *Dilute Hydrochloric Acid* — 1 : 3 (*v/v*).

4.2.12 *Ammonium Chloride Solution* — 5 percent.

4.2.13 *Hydrofluoric Acid* — 40 percent.

4.2.14 *Standard Iodine Solution* (1 ml = 0·000 15 g of As) — 0·01 N. Dissolve 1·27 g of re-sublimed iodine and 4 g of potassium iodide in 25 ml of water. When the solution is complete, dilute to one litre with water and store in a dark-coloured glass-stoppered bottle. Standardize against standard arsenious oxide solution.

4.2.15 *Standard Arsenious Oxide Solution* — 0·01 N. Dissolve 0·495 g of arsenious oxide in about 4 ml of sodium hydroxide solution (10 percent), and dilute to about 200 ml. Add dilute hydrochloric acid (1 : 20) until just acidic. Add about two grams of sodium bicarbonate and dilute to one litre in a volumetric flask.

4.2.16 *Starch Solution* — Make a suspension of one gram of soluble starch in about 10 ml of water and add it carefully to 100 ml of boiling water. Boil for two to three minutes and cool. Prepare the solution fresh as needed.

4.2.17 *Sodium Bicarbonate* — (carbonate-free) solid.

4.3 Procedure

4.3.1 Weigh accurately 5 g of the sample and 0·5 g of electrolytic copper, transfer them to a 600-ml squat beaker, and dissolve in a mixture* of 30 ml of dilute sulphuric acid, 15 ml of concentrated nitric acid and 20 ml of concentrated hydrochloric acid. If a large number of determinations have to be carried out, use 200 ml of the mixed acids (*see* **4.2.6**) for each determination.

4.3.2 Dilute the mixture slightly with hot water and filter off any graphite. Wash the residue with hot dilute nitric acid and discard it. To the filtrate add 0·5 g of potassium permanganate and boil the mixture for at least 5 minutes. Add enough sulphurous acid solution dropwise till the filtrate is clear.

4.3.3 Evaporate the solution to fumes and heat until all the nitric acid is expelled. Add about 75 ml of hot water and boil. (In case of high silicon iron, silica should be filtered off at this stage.) Concentrate the filtrate to 75 ml and transfer to 750-ml conical flask, washing the beaker with 75 ml of concentrated hydrochloric acid. Add 2 g of sodium hypophosphite and warm to brisk effervescence avoiding boiling. Add

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*In case of tungsten steel, mixed acids (*see* **4.2.6**) only shall be used for the dissolution of the sample.

by instalments 2-g portions of hypophosphite till effervescence ceases and finally add 12 g of sodium hypophosphite. Insert a cork carrying a reflux condenser tube (600 × 10 mm) in the mouth of the conical flask and boil the contents of the flask for 15 minutes. Cool the solution and filter through a pad of pulp prepared in accordance with **3.1**. Wash the arsenic precipitate with 100 ml of dilute hydrochloric acid containing 3 g of sodium hypophosphite and then 7 times with ammonium chloride solution. Further proceed in accordance with **4.3.5** and **4.3.6**.

4.3.4 *Procedure for Reduction of Arsenic Acid (in Case of Tungsten Steel)* — Transfer the clear solution from **4.3.2** to a quartz flask, and add 40 ml of water. Boil the solution to expel nitric acid fumes completely; cool, add 80 ml of concentrated hydrochloric acid and 40 ml of water. Add 2 g of sodium hypophosphite, warm to brisk effervescence avoiding boiling, and add by instalments 2-g portions of hypophosphite till effervescence ceases. Add 10 drops of hydrofluoric acid and 12 g of sodium hypophosphite. Boil under reflux tube and filter and wash the reduced arsenic as described under **4.3.3**.

4.3.5 Discard the filtrate and transfer the residue along with the filter pad to an 800-ml tall-form beaker, rinsing the funnel with 50 ml of water. Run in a measured excess of the standard iodine solution (about 30 ml are usually adequate). Stir to disintegrate the filter pad, allow to stand for 5 minutes and dilute to 250 ml. Titrate with the standard arsenious oxide solution to pronounced lightening of the iodine colour. Add a few ml of starch solution; discharge the coloration by adding about 3 ml more of standard arsenious oxide solution and about 2 g of sodium bicarbonate. Back-titrate cautiously with the standard iodine solution, shaking vigorously for each addition, until the blue colour just re-appears.

4.3.6 Carry out a blank determination following through all steps of the procedure and using the same amounts of reagents (including 10 ml of the standard arsenious oxide solution) but without the material. Since arsenic is oxidized from the elemental state, to the pentavalent state, therefore 10 ml of standard arsenious oxide solution used in blank estimation represent 25 ml in the final titration. Any amount in excess of this 25 ml that is required in titrating the blank represents volume of the standard iodine solution required for the blank.

4.4 Calculation

$$\text{Arsenic, percent} = \frac{[(A - B) - C] \times 0{\cdot}000\,15}{M} \times 100$$

where

A = total volume in ml of the standard iodine solution added in the test,

B = volume in ml of the standard iodine solution required for the blank,

C = total volume in ml of the standard arsenious oxide solution used to titrate the excess iodine solution, and

M = mass in g of the sample taken.

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